

CENTRAL INTELLIGENCE AGENCY

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1. Introduction

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- b. Unit for Heavy Water Production by Fractional Distillation
- c. Unit for Heavy Water Production by Isotope Exchange at Normal Pressure
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a. Unit known as "Stalin's Organ" for Heavy Water Production

- (1) Although I never saw or even heard of this unit while at Leuna, Dr Hans Offe, now at Leverkusen, referred to it in a conversation during the Christmas Holidays of 1951. He remembered having seen this unit in the fall of 1945, standing in the southwest corner of Leuna Bldg ME 225.
- (2) The unit consisted of an upright sheet iron cylinder, about one meter in diameter and 2.5 meters high, from the top of which protruded a large number of vertical sections of glass tubing, arranged concentrically around a cylindrical core, thus giving a general pipe organ effect. He told me that he thought at that time it might have been used for the electrolytic production of heavy water, but observed neither rectifiers nor electrolyte.
- (3) Dr Walter Schmidt never saw this unit, but recalled a slogan, "Hundred-fold vaporization and condensation". It is possible that this slogan applied to this unit, which may have been used to obtain experimental data for the design of the Bitterfeld column, whose function was to separate heavy water by fractional distillation.
- (4) In 1947, a girl laboratory assistant, whose first name was Nadua and who worked closely with Prof Borleskov on heavy water projects, was sent on a mission to a place about midway between Moscow and Leningrad. There is a very remote possibility that "Stalin's Organ" was reinstalled there.

b. Unit for Heavy Water Production by Fractional Distillation

- (1) During the war years of 1943-44, a column for the separation of heavy water from ordinary water by fractional distillation was designed at Leuna by Dr Adalbert Orlicek, now a lecturer at the University of Vienna. I do not know for what enrichment this unit was planned, but the design called for an atmospheric column 100 meters high, containing an unknown number of wooden grids, which were chosen in preference to bubble caps or perforated plates in order to minimize the resistance to flow. I never saw this unit, but assume the column was to be one - two meters in diameter.
- (2) Because of its height, this column was to be broken into four 25-meter sections, installed in a row. These sections were fabricated at Leuna, but due to air raid hazards were sent to Bitterfeld to be erected there. Dr Karl Geib and his laboratory were also evacuated to Bitterfeld to operate the column. I do not know if the installation was ever completed, but I do know the column never produced and that at the end of the war it was dismantled and removed by the Soviets. I am not certain as to its final disposition, but the evidence would indicate one section was erected at the Karpov Institute.

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[REDACTED] a column, [REDACTED] in diameter and 25 meters high, erected in the stairwell of the east wing of the lower of the two main buildings comprising the Karpov Institute. Drs Herold and Gemassner thought it looked like one of the sections of the Bitterfeld column. Within a few days, however, before Dr Geib and his group arrived, the column was hidden from sight by a plywood partition which filled the entire stairwell, and only the upper portion, which extended about five meters above the roof, was still visible from the street.

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- (4) This evidence was supported by the Soviets' interest in wetted-wall columns, which despite their lower efficiency, have even less resistance to flow than wooden grid columns, and one of our first assignments was to draw up plans for a wetted-wall column constructed from concentric cylindrical pipes. [redacted] started this work, but were in [redacted] assignments.

c. Unit for Heavy Water Production by Isotope Exchange at Normal Pressure

- (1) The production of heavy water from ordinary water by catalyzed isotope exchange at normal pressure was studied at Leuna during the war years by Dr Karl Geib. After his laboratory work had reached the pilot plant stage, Dr Heinrich Elm was made his assistant.
- (2) The pilot plant was installed in Leuna Bldg ME 263. I saw it only once or twice. It consisted of five - six heavily insulated rectangular stages, separated by preheaters and coolers. Each stage contained two catalyst contact chambers packed with standard Leuna hydrogenation catalyst, formula $3\text{Ni} \cdot \text{Al}_2\text{O}_3$, and operated at 100°C and 700°C respectively.
- (3) The theory of separation was based on the fact that for the reaction $\text{H}_2\text{O} + \text{HD} = \text{HDO} + \text{H}_2$ the equilibrium constant varies from 2.02 at 100°C , to 1.07 at 700°C , and thus by alternate operation at these temperature levels, carried on countercurrently in a series of stages, enrichment should be achieved.
- (4) This plant was operated for the Soviets after the war. The enriched product, containing 1% heavy water, was sent to Moscow. Production was small. From July to October 1946, I recall only one 50-liter flask being shipped. In about November 1946, this plant was dismantled and removed by the Soviets. 25X1X
- (5) At the Karpov Institute, [redacted] were turned over to Prof Borleskov. One day a former laboratory assistant, Nadua, on a visit to the Institute, mentioned working for Prof Borleskov in a laboratory located in the northern outskirts of Moscow.

[redacted] sible that the Leuna plant was reassembled there.

d. Unit for Heavy Water Production by Isotope Exchange at 700 atmospheres

- (1) The Soviets were dissatisfied with the separation factor, as well as the high temperature level, and the large amount of heating and cooling involved in the normal pressure unit for heavy water production by catalyzed isotope exchange. Early in 1946 they requested Dr Karl Geib to investigate the effect of pressure on the equilibrium constant. Dr Geib was skeptical of results, but began the construction of an experimental column, about two - three inches in diameter and eight meters high to obtain data at 700 atm. This pressure was chosen simply because of Leuna experience at that pressure.
- (2) The column was erected in Leuna Bldg ME 499. Installation was almost finished when the SMA research group left in October 1946. I later learned it was completed in November 1946, leak tested, and, before a run could be made, dismantled and taken to the USSR, together with the normal pressure installation.

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- (3) The 700 atm column was brought to the Karpov Institute, and its re-assembly was started in March or April 1947. It was to be installed in a separate brick building, 10 by 15 meters, and 15 meters high, located in the southeast corner of the Karpov Institute grounds.

- (4) [REDACTED] the column, which was made up of six sections, had not yet been fitted, and was lying along the south wall, in front of the six-stage compressor installed at that end of the building. An instrument panel frame had already been erected, but measuring instruments were not in evidence. Electrolyzers, for the production of hydrogen, were mounted on a balcony frame, reached by a six-meter ladder, on the east wall of the building. A hydrogen gas storage tank, 10 meters in diameter and eight meters high, with hydraulic seal, was being erected just west of the building. When we left the Institute in July 1948, a wooden partition was being constructed around the gas storage tank.

e. Catalyst Shipment to Norway in 1942-43

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Several hundred liters of a nickel catalyst, designated SH200, were shipped to Norway. This catalyst was not a standard Leuna catalyst. All he knew concerning its preparation was that it involved soaking an inert material in nickel nitrate solution. He was uninformed as to its disposition in Norway, and whether or not it was later returned to Germany.

f. Catalyst for Heavy Water Production by Isotope Exchange at Normal Pressure

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[REDACTED]

at normal pressure. The catalyst first tried in the pilot plant at Leuna had been the standard hydrogenation catalyst, consisting of $3\text{Ni} \cdot 1\text{Al}_2\text{O}_3$, used in the production of cyclohexanol from phenol. It had the serious defect of poor mechanical strength, and formed a slurry after prolonged agitation by the mixed stream of water vapor and hydrogen in the contact chamber. Recalling the exceptional strength of the Leuna oil cracking catalyst, composed of $1\text{Al}_2\text{O}_3 \cdot 9 \text{SiO}_2$, I reasoned that the addition of some silica to the hydrogenation catalyst might appreciably increase its strength without too great a sacrifice in activity. Knowing that the activity is decreased by silica, [REDACTED] and found that the mechanical strength of alumina increased rapidly with additions of silica up to 5%, then slowly up to 10%, after which the strength hardly varied. It therefore appeared that about 95% alumina and 5% silica might represent the optimum composition for the nickel catalyst support. Thus guided, a number of catalysts were prepared as follows: black nickel oxide, which is a mixture of NiO and Ni_2O_3 obtained by igniting nickel carbonate, was mixed with the alumina-silica support in the dough stage, which was then extruded, formed, and dried as six mm spheres. The nickel was then reduced by an eight-hour exposure to hydrogen, at 400°C , using 1000 volumes of hydrogen per hour per volume of catalyst. The finished catalyst was tested for mechanical strength by a simple crushing test, and by rotating the catalyst for 24 hours in a bottle, and noting the powder formed.

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- (2) In the beginning, Drs Geib and Elm were supposed to test the catalysts for activity in the following manner: A charge of about 50 cc of catalyst was placed on a support in an upright, jacketed, condenser column, and maintained at 100°C by condensing steam in the jacket. Hydrogen, from the laboratory line, was bubbled continuously through a charge of water in the flask directly under the column, and the mixture, whose composition was controlled by the water temperature, was passed over the catalyst charge, and then through a condenser from which the hydrogen was burned or discarded, and the water recycled to the flask, or removed for testing. The heavy water concentration was determined by a graduated series of calibrated quartz floats, about five - eight mm in diameter, which had to float submerged half way between the top and bottom of the liquid. The temperature was held exactly at 20°C by a Hoeppler thermostat with a temperature control sensitivity of $\pm 0.01^\circ\text{C}$.

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- (3) [REDACTED] prepared at the Karpov Institute were tested by the Soviets in a similar manner. About 20 catalysts for isotope exchange at normal pressure were delivered to Prof Borieskov for testing, but I do not know the results.

g. Catalyst for Heavy Water Production by Isotope Exchange at 700 atmospheres

- (1) At a pressure of 700 atm a porous catalyst is probably not necessary, and I worked on the basis of a mechanically strong catalyst support with an active nickel coating.
- (2) One catalyst was prepared from nickel wire spirals five - six mm in diameter, which were oxidized and reduced three or four times to give an active surface. Another catalyst was made by treating porcelain chips, six - seven mm in diameter, with nickel nitrate and then igniting and reducing to give an active nickel surface. A third catalyst was a Raney nickel catalyst, with only a small amount of the aluminum dissolved out with caustic, thus leaving the catalyst mechanically stronger.

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(3)

[REDACTED]

add here that the catalyst spheres were formed at Karpov Institute by a small hand-operated machine made by the firm Franke in Leipzig. This machine had been repaired from the Leuna Research Laboratory. The standard Leuna pelleting machines were mechanically operated. They were also produced at Franke, Leipzig, bearing the trade name Francoma. These machines have a capacity of 200 - 300 liters/hr each of catalyst pellets.

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h. Planned Catalyst Production Plant at Chirchik, near Tashkent

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[REDACTED]

be small in size and was designed to produce nickel-alumina catalysts of the $5\text{Ni} \cdot 1\text{Al}_2\text{O}_3$ variety, which had been used at Leuna for hydrogenation of phenol to cyclohexanol as well as for heavy water. I would roughly estimate the capacity, which was not shown on the plans, to be 10 - 20 tons/month of finished reduced catalysts, if the plant worked in shifts. I do not know who drew up the plans.

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- (2) It appeared that the plant was intended purely for production, that the stone building was ready, but that equipment had not yet been installed. The plant was designed entirely after the Leuna pattern and from the number of times Prof Borieskov asked me if the Leuna method agreed with the plans, I consider it quite possible that dismantled Leuna equipment was to be installed there. The one difference between the Leuna method and the Chirohik method was that the Leuna production was vertical while the Chirohik plans showed horizontal production.
- (3) I do not know where the raw materials came from, but am almost certain they included metallic nickel, metallic aluminum, and sodium hydroxide. I recall definitely that the plans included mixers, filter-presses, ball mills, and a press to make the pellets, but I do not remember

that hydrogen was piped to the roof and burned off, but such piping would be necessary to accommodate the hydrogen arising from the digestion of metallic nickel in nitric acid and the metallic aluminum in sodium hydroxide. It is therefore possible that the finished product was to be unreduced catalyst, corresponding to Leuna No 3390, rather than the final reduced catalyst corresponding to Leuna No 6523.

- (4) The production of unreduced catalyst would suggest either of the following:
- (a) The plant where the catalyst is to be used has facilities for reducing the catalyst, or
 - (b) The catalyst is to be shipped a long distance to its destination, in which case unreduced catalyst would be sent since reduced catalyst is very sensitive to atmospheric influences.

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1. Hydrazine Hydrate, Hydrogen Peroxide, and Amines for Rocket Fuels

- (1) Hydrazine hydrate was worked on at Leuna during the war. There was no evidence of this work at Leuna in 1946. However, the Siebel Plant at Halle worked on hydrazine hydrate as late as 1946 when in October, her scientists were taken to Kalinin, north of Moscow. Dr Emerick, Chief of the Siebel Plant, was head of this group in the USSR.
- (2) Hydrogen peroxide was developed at Leuna during the war for use as a rocket fuel. It was probably also used with submarine diesel engines to eliminate bubbler in the wake. Hydrogen peroxide was made by the calcium dioxide method under Dr Freshlich, and by the propane peroxide method under Dr Jochinke. I have no knowledge of hydrogen peroxide manufacture in the USSR but believe there is a nitric acid plant at Severo-Donetsk.
- (3) Ethyl amines were prepared by Dr Andreas in the basement of the Kar-pov Institute, where he did research in a bench scale oven reactor which had been dismantled and brought from Leuna. The reactor had a capacity of 200 - 300 cc of catalyst. The ethyl amines were prepared from ethyl alcohol and ammonia at 15 - 20 atm pressure, over alumina and kaolin, which are the standard dehydration catalysts for the production of methyl amines. Because of the poor rocket-fuel characteristics of mono- and triethyl amine, only the diethyl amine was desired. It was suggested that pressures of 250 - 300 atm might favor the formation of the diethyl amine.

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- (4) A cyclic amine, which had been described in Chem Helvetica a number of years before the war, was found by Dr Asinger to be an excellent rocket fuel with good ignition qualities. It was prepared by reacting acetone with ammonia in the presence of a catalyst. Dr Asinger had worked with manganese, iron, and cobalt salts as catalysts in the preparation of various amines, and found that cobalt acetate was the best catalyst in the preparation of this particular amine.

j. Pilot Plant for Rocket Fuel Production at Antonovka, near Moscow

- (1) The Soviets were so interested in the cyclic amine prepared by Dr Asinger from acetone and ammonia using cobalt acetate as catalyst, that they requested he build a pilot plant for its production. This plant, when finished by Asinger, was installed in plant 54 or 56 in a factory several hundred meters north of the railroad station in Antonovka, a suburb southeast of Moscow. Asinger was permitted to see the pilot plant once after it was installed, but was not permitted to enter the main plant.
- (2) The rocket fuels group at Karpov Institute said they believed there was a rocket testing field nearby where the Soviets tested the fuels produced by the group.

k. Possible Plant for Rocket Fuel Production at Dzerzhinsk

- (1) When the German scientists were transferred from Karpov Institute, Drs Asinger, Froehlich, Jochinke, Andreas, Scheuer, and Elm were sent to Dzerzhinsk where they continued research on rocket fuels with Asinger in charge. Apparently, there is a plant at Dzerzhinsk for the production of rocket fuels, and it seems likely that the amine plant which was removed from Leuna was brought to this place.)

l. Working Conditions at the Karpov Institute

- (1) Working conditions at the Karpov Institute were not pleasant. The laboratories were small and crowded by the presence of two scientists with their service personnel. Chemicals were scarce. The storage rooms serving the entire institute consisted of two small rooms, about four by six m each, with shelves along the walls. The quantity of chemicals stored there would correspond to the quantity designed to supply a small test laboratory at Leuna, yet the Karpov Institute was staffed with about 100 persons, and was the Research Institute of the Ministry for Chemical Industry. The scarcest items were the purest chemicals of analytical grade. They were only issued by grams, far below the requested quantities. Sodium hydroxide for titration, for instance, could only be obtained with the special permission of Prof Borieskov, and at a maximum quantity of 50 grams, which was generally far short of our requests. It was imported from

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copper, and brass, and could be used only after a time-consuming purification. Neither the shortage of materials, nor the untidy working methods improved as long as we stayed at the Karpov Institute.

- (3) Frequent breakdowns of the electric current were annoying. They were particularly dangerous when we were making our nickel nitrates and had the containers with the nitrore gases standing under the electrical ventilation system. The unexpected breakdowns of the electrical system caused poisonous gases to fill the room, endangering our lives.
- (4) All our written work was classified Top Secret (SS in Russian). Disclosures were punished by ten years of hard labor. All our notes had to be written in laboratory journals, the pages of which were numbered before they were issued and tied together with a sealed cord. Every evening the journals were collected and handed, together with the laboratory keys, to Prof Borishev. Checks on the journals were made at irregular intervals by the First Department of the Institute, to check if all pages were still in their proper places. When the journals were finished, they were collected by the First Department and kept in its files. In the evening, at the close of the shop, the doors of the laboratories were locked and sealed on the outside. The night guards did not dare break this seal, even when they saw (as happened twice in my laboratory) that the water faucet was not shut off, and that the laboratory was being flooded.
- (5) The library of the Karpov Institute was not very well stocked. It was located in the "Upper Korpus" on the first floor. There was another library in the "Lower Korpus"; this was not officially the library of the Karpov Institute but the library of the Chemical Ministry, and seemed quite deficient in material for a ministerial library. A good library and archive existed in the GIAP. Most of the Leuna literature on nitrogen production was kept there. In contrast to the Karpov Institute, the archives of the GIAP could be used by the Germans. The archive was kept in the basement of the old building. It contained the so-called "doctor archive" which consisted of all the material found in the desks of the Leuna scientists who were deported to the USSR. When the scientists were transported out of Leuna, their desks were sealed by the Soviets and shipped intact to the GIAP in Moscow. There, their contents were removed and incorporated in the "doctor archive".

m. Instrument for Measuring Ignition Period of Rocket Fuels

- (1) Dr Scheuer, assisted by Dr Elm, after the latter had been requested to give up his work on heavy water, assembled an apparatus for measuring the ignition delay of rocket fuels as follows: The amine to be tested as a rocket fuel was dropped through a beam of light into a dish of 98% nitric acid. The light beam fell on an oscillating reflector and was reflected onto a photoelectric cell attached to an oscilloscope. The passage of the drop through the beam, and the resulting flash of the amine in the dish of nitric acid, appear as interruptions in the vibrating line on the screen of the oscilloscope. A moving photographic film records all that appears on the screen. ^{25X1A} From a study of this film the time elapsing between the two interruptions, and also the time of the ignition delay, can be calculated to

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